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APPLICATION OF FAST NEUTRON ACTIVATION ANALYSIS TO POLYMER DOSIMETRY

by

Arthur D. Coates
Joe A. Swisher

March 1966

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APPLICATION OF FAST NEUTRON ACTIVATION ANALYSIS TO POLYMER DOSIMETRY

Arthur D. Coates
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Aberdeen Proving Ground, Md.
March 1966

APPLICATION OF FAST NEUTRON ACTIVATION ANALYSIS TO POLYMER DOSIMETRY

ABSTRACT

Fast neutron activation analysis was used to measure the gamma dose absorbed by a polymer. A series of low density polyethylene films was irradiated by cobalt-60 gamma rays in the presence of air. The gamma doses ranged from 1×10^5 to 4.7×10^7 rads. The oxygen content of the polymer films increased with increasing dose. The increase in oxygen content of each film was monitored at 1720 cm^{-1} by infrared spectroscopy. The oxygen increase also was monitored by measuring the activation of oxygen-16. This latter method employs fast neutron activation of oxygen-16 and the measurement of the emitted radiation to determine gamma dose. The results of both methods of dosimetry are presented, and the improvements offered by the neutron activation technique are indicated.

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INTRODUCTION

This report presents the results of a study on the application of fast neutron activation analysis to high level gamma ray dosimetry. The need for high level dosimetry has become greater with the advent of increasingly larger irradiation devices which emit gamma doses greater than 1×10^6 rads in less than one hour. Previous studies by the Ballistic Research Laboratories utilized the radiation induced chemical changes in polyethylene to determine gamma ray dose by infrared spectral techniques.^{1*} This report discusses a method for extending the range of polymer dosimetry above 5×10^7 rads and for improving the accuracy at lower dose levels. The method employs fast neutron activation of oxygen-16 and the measurement of radiation emitted during the decay processes for determining the gamma ray dose. Both methods of dosimetry are based upon the finding that the oxygen content of the low density polyethylene, when irradiated in air, increases with gamma ray dose.

Several chemical reactions occur within polyethylene when this material is exposed in air to gamma radiation.² These reactions include crosslinking, formation of random double bonds, hydrogen evolution, main and side chain cleavage and degradative oxidation.

Oxygen reacts with polyethylene when the latter is undergoing gamma irradiation, and forms several carbonyl and hydroxyl groups. The increase in oxygen content of polyethylene is observed spectrally at three different wavelength regions. The spectral regions are listed below:

3300 - 3400 cm^{-1}	Hydroxyl Groups
1600 - 1800 cm^{-1}	Carbonyl Groups
800 - 1200 cm^{-1}	Fingerprint Region

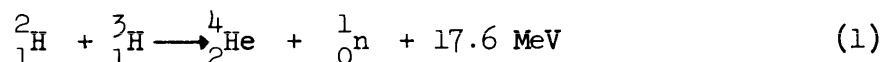
As the oxygen content of polyethylene increases, the spectral absorption increases. This change in absorption has been correlated with dose and is the basis of the technique for utilizing polyethylene film as a gamma ray detector. The use of infrared spectral techniques is a convenient and reasonably sensitive method for observing the increase in oxygen content

* Superscript numbers denote references which may be found on page 19.

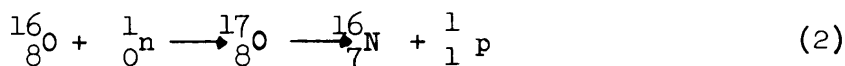
as a function of gamma dose. Figure 1 shows the change in spectral absorption at 1720 cm^{-1} in gamma irradiated polyethylene versus gamma dose.

Published reports³⁻⁶ have shown that fast neutron activation is a rapid and sensitive method for determining oxygen-16. The minimum detectable limits are reported to be 10 parts per million (ppm) in samples containing 10 mg or more of oxygen.⁴ The extension of fast neutron activation analysis to polymer dosimetry, by utilizing the oxygen-16 contained in irradiated hydrocarbon polymers, should reduce the time required for determination of dose, particularly where large numbers of individual measurements are to be performed. In addition, it may be possible to extend the upper and lower limits of the useful range, and conceivably to improve the accuracy of the polymer dosimetry technique. The threshold energy required for neutron induced activation of oxygen-16 is 9.6 MeV.³ The abundance of oxygen-16 in oxygen is 99.76 percent.⁷

It is convenient and reasonably inexpensive to obtain 14.3 MeV neutrons by using a Cockcroft-Walton type accelerator to accelerate deuterons to approximately 150 KeV. The deuterons then strike a tritium target.⁸ The activation studies reported herein were performed with a system of this type. The fast neutrons are produced by the reaction:



The concentration of oxygen-16 in the irradiated polymer was determined from the quantity of radiation emitted by the decay of nitrogen-16. The half life of nitrogen-16 is 7.4 seconds. The reactions that occur during the activation of oxygen-16 are:



The disintegration scheme for nitrogen-16 is shown in Figure 2.

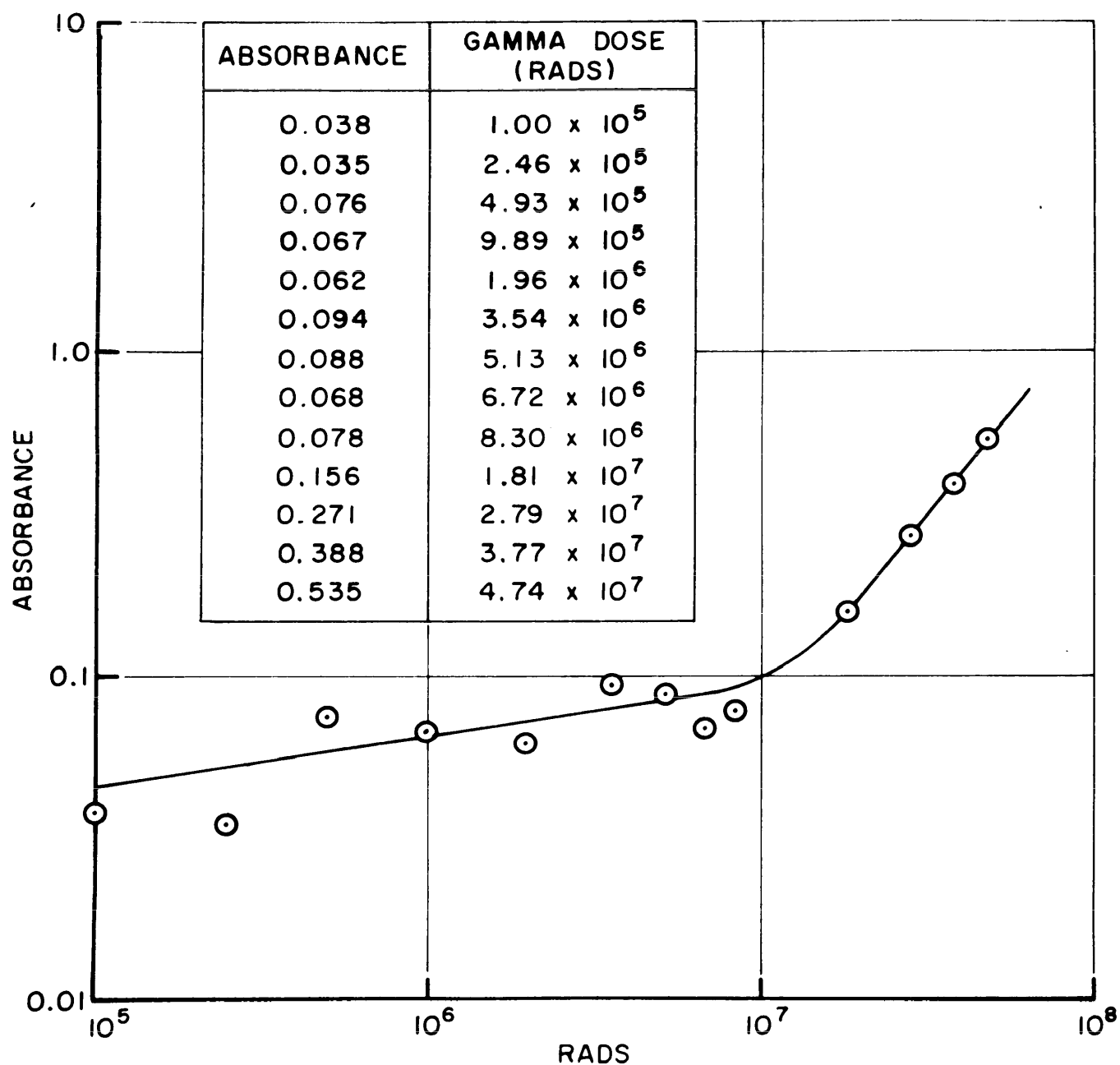


FIGURE 1. THE CHANGE IN ABSORBANCE AT 1720 CM^{-1} IN POLYETHYLENE VS. GAMMA DOSE.

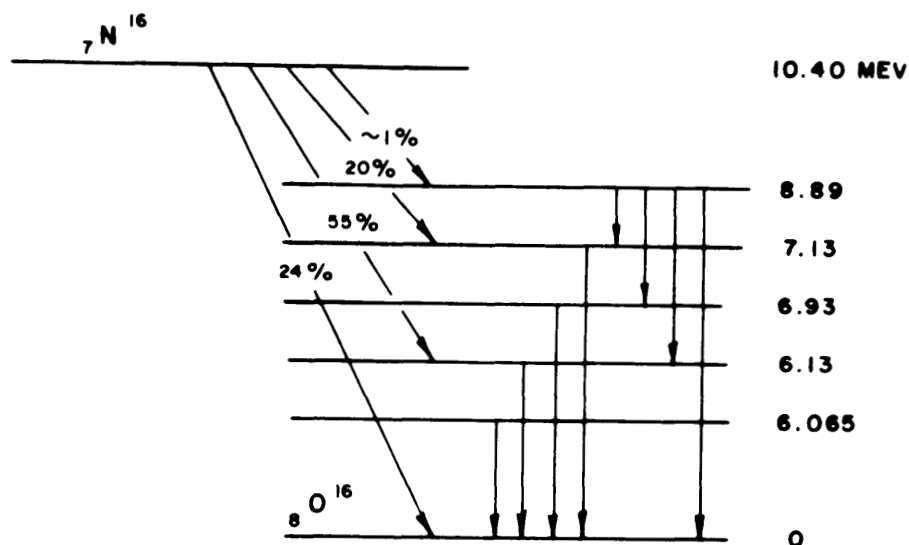


FIGURE 2 — NITROGEN-16 DISINTEGRATION SCHEME.⁹

Reaction 3 is the basis for the analytical determination, since the quantity of the emitted gamma radiation is proportional to the oxygen concentration. This gamma radiation is detected and measured by scintillation spectrometry. The normalized activity data obtained from each activated polymer sample is then related to the known gamma dose absorbed previously by the respective polymer films in a high intensity gamma irradiation unit. The normalized counts are then plotted versus gamma dose; these provide the instrument calibration in counts as a function of gamma dose.

The specific oxygen content of each irradiated polymer film may be determined if desired. This is accomplished by relating the normalized counts from each activated sample with the data obtained in an identical manner from standard samples of known oxygen content using the relationship:

$$WT_x = \frac{A_x \times WT_o}{A_o} \quad (4)$$

where:

A_o = total counts from oxygen standard

A_x = total counts from unknown sample

WT_o = weight of oxygen in standard sample

WT_x = weight of oxygen in unknown sample

The weight of oxygen in each irradiated sample may be determined, and related to gamma dose received by the polymer film. This provides a method for determining gamma ray dose and has potential use in inter-laboratory comparisons where different types of radiation counting equipment are employed. The use of the oxygen weight characteristic also would permit laboratories without activation facilities to conduct polymer dosimetry studies by employing techniques such as the classical Unterzaucher method for oxygen determination.

It should be noted, that in the use of low density polyethylene for gamma dosimetry, each lot of polyethylene studied by BRL has required gamma dose calibration. Therefore, the oxygen content data should be considered typical only for a specific type and lot of polyethylene film.

EXPERIMENTAL PROCEDURES AND APPARATUS

The polyethylene film used in this dosimetry study was supplied by the E. I. DuPont De Nemours and Company on special request. The film had a uniform thickness of 2.0 ± 0.2 mils and a density range between 0.915

and 0.925 gm/cm^3 . The samples were prepared in strips 1.9 cm wide by 122 cm in length and weighed prior to irradiation. The infrared spectrum of each polymer film also was obtained prior to irradiation. The sample films were then placed in an open container, irradiated and subsequently reweighed.

The gamma irradiations were performed in the BRL high intensity gamma irradiator, which is a version of the AECL Gammacell system. The gamma dose in the irradiation cavity of this cobalt-60 unit was determined by the manufacturer and verified by these Laboratories. The calibrations and verifications were performed using the Fricke chemical dosimeter.¹⁰ Isodose curves for each irradiation position in the cavity were established. The dose rate at the center of the cavity during the polymer exposures was $1.08 \times 10^6 \text{ rads/hr} \pm 3.5 \text{ percent}$.

Following the irradiation, an infrared spectrum was obtained of each polyethylene film. The change in spectral absorption at 1720 cm^{-1} also was obtained in order to monitor the oxygen increase attributable to carbonyl linkages. These measurements were obtained using the Perkin-Elmer Corporation Model 13 double beam, ratio recording spectrophotometer equipped with sodium chloride optics and densitometer attachment.

Each polymer film was then wrapped as a tight concentric unit and placed in an activation sample container in preparation for the fast neutron activation analysis. The polyethylene sample containers have been described elsewhere.⁸

The activation analysis of each of the gamma irradiated films was performed at the Oak Ridge National Laboratories. A Texas Nuclear Corporation Model 9501, 150 KV Cockcroft-Walton type generator was used. The beam current was 500 μa and the target was tritiated titanium. A Radiation Instrument Development Laboratories 400 channel analyzer with an electric typewriter printout system was used in conjunction with two $3" \times 3" \text{ NaI(Tl)}$ scintillation crystals, a rapid transfer system for the activated sample and a lead shielded sample counting chamber.⁸

In these experiments the fast neutron bombardment of the sample had a duration of ten seconds, the transfer time with preset counting delay was three seconds and the counts from the sample were accumulated for thirty seconds which is approximately four half-lives of nitrogen-16. The lower and upper level discriminators of the multichannel analyzer were adjusted for optimum counting of the high energy gammas, and minimizing the counts from interfering reactions.

In order to compare gamma dose with the specific oxygen-16 content of the polymer films, samples of silicon dioxide were used as oxygen standards. The oxygen content of each irradiated film was determined by comparison of the activity of the gamma irradiated film with the activity of the oxygen standards.

RESULTS AND DISCUSSION

The activity data obtained using fast neutron activation analysis in polymer dosimetry are presented in Figure 3. The neutron induced activity of each polymer film is plotted as normalized counts obtained from each sample versus gamma dose. The activity measured in each sample is attributable primarily to the radioactive decay of nitrogen-16 to stable oxygen-16. The quantity of radiation detected represents the total integrated counts obtained over the range of 1.7 to 9.7 MeV, which includes the 6.13 and 7.13 MeV gamma photopeaks of nitrogen-16. Using the techniques described herein, no significant interfering reactions were found in the neutron activated polymer films. Each activated film decayed following the half-life pattern of $^{16}_7\text{N}$.

The results show that the greater the gamma dose received by the polymer film, the larger the total number of counts detected subsequently in the neutron activated polymer film. This indicates that the oxygen-16 content of the polymer films increases with gamma dose, since it has been established that the number of counts obtained from the activated oxygen compounds are proportional to oxygen concentration. It is known that polyethylene irradiated in air by gamma rays contains radiation induced quantities of oxygen.¹¹ Figure 4 shows the percentage of oxygen present in irradiated polyethylene as determined by neutron activation.

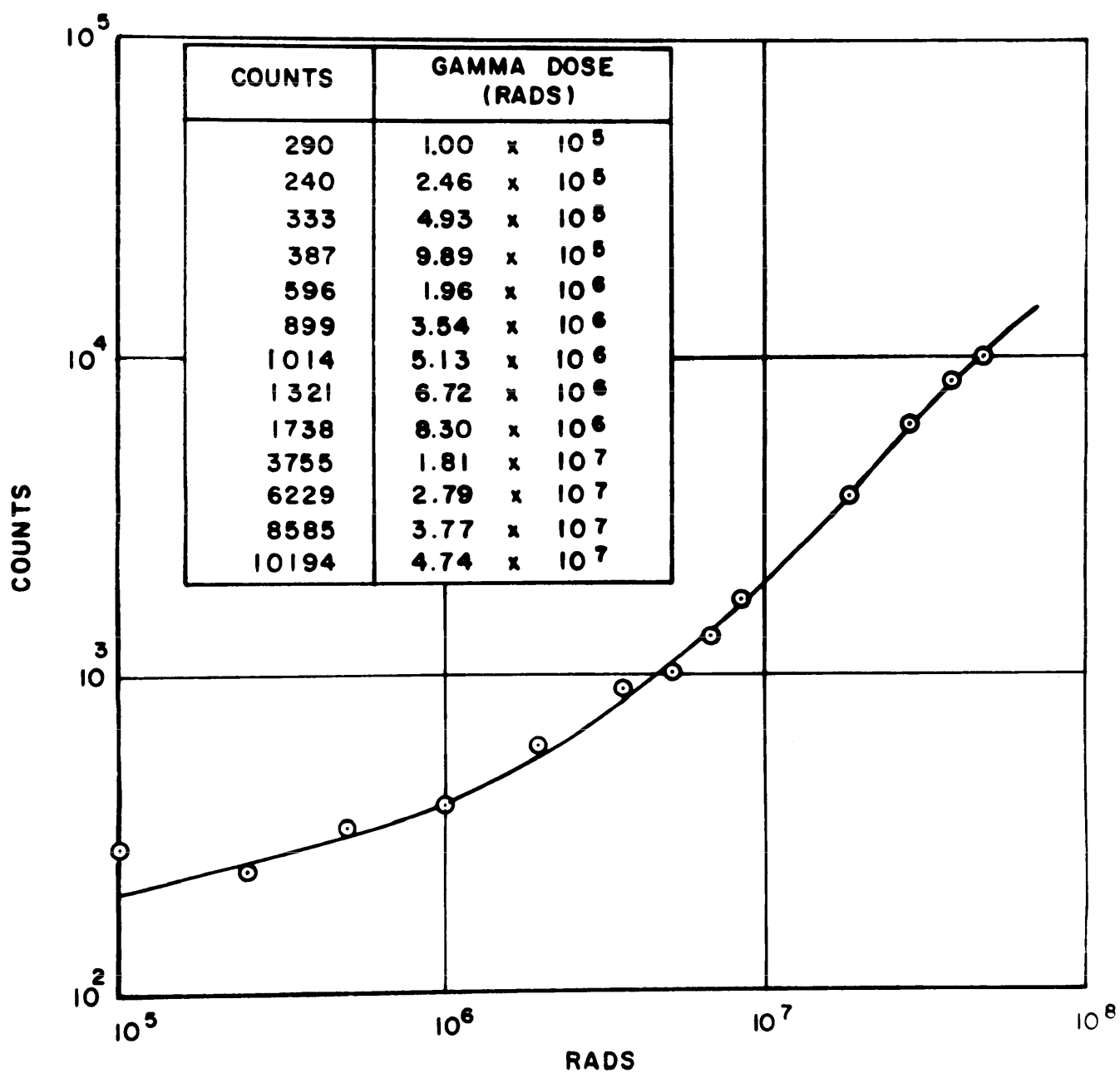


FIGURE 3. THE COUNTS OBTAINED FROM ACTIVATED POLYETHYLENE SAMPLES VS. GAMMA DOSE.

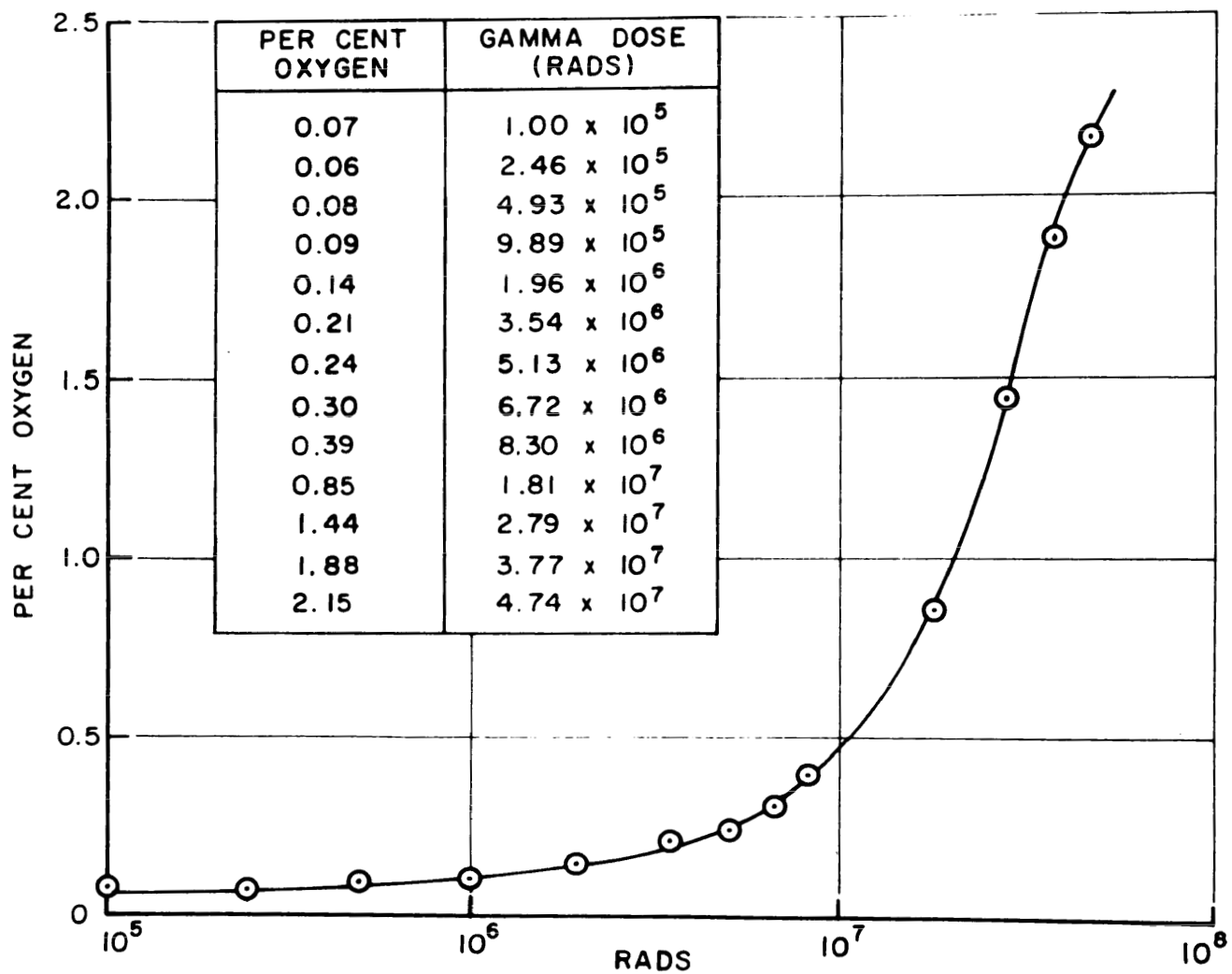


FIGURE 4. RADIATION INDUCED OXYGEN CONTENT OF POLYETHYLENE VS. GAMMA DOSE.

Each gamma irradiated polymer film was studied by infrared analysis prior to neutron activation. Figure 1 presented earlier shows the increase in absorption at 1720 cm^{-1} versus gamma dose, this change in absorption is attributable to radiation induced C=O formation in the polyethylene. The technique for relating dose with infrared absorption was reported previously, and is included here for comparison.

Polymeric films of the olefin type offer several advantages for use in dosimetry studies in the megarad range. Low density polyethylene in particular, has been found to be useful because of its relatively high degree of stability, minimum significance of post irradiation reactions, and simplified infrared spectrum. Dosimetry measurements with polyethylene films utilizing either activation analysis or infrared spectral techniques possess several desirable characteristics. The advantages include: (1) the time required for dose determinations by either method is less than five minutes; (2) the irradiated polymer detector may be analyzed at a given time and verified if desired at a later date; (3) the gamma dose to the detector may be provided in either single or multiple exposures; (4) the use of lengthy analytical procedures and highly purified compounds is not necessary and (5) the data obtained are relatively simple to resolve by utilizing predetermined calibration curves. In addition the data indicate that the polymer neutron activation technique has the added advantage of increased accuracy and extended dose range.

Figures 1 and 4 show respectively the infrared and activation dosimetry data using the same irradiated polymer film for both determinations. Over the dose range studied, it is observed that the experimental points on the activation plot show less random deviation than those of the infrared curve.

With either technique, the polymer films of the type used in these experiments have a lower usable dose limit in the range of 1×10^5 rads. This limit results from sensitivity characteristics of the instrumentation, and not from the inherent characteristics of the polymer films. The data shown in Figure 3 indicate that the range may be extended by using activation analysis.

With respect to higher dose level determinations, the infrared technique is limited to measurable optical density changes. At 1720 cm^{-1} the polyethylene film used here approaches an optical density of infinity at dose levels of approximately 1×10^8 rads. Polyethylene film exposed to this dose level has begun to deteriorate physically and has become brittle. Thus, the determination of absorbed dose becomes increasingly difficult. Therefore, the analytical sensitivity and the physical condition of the polymer limit the upper usable dose range of the infrared technique.

Fast neutron analysis of the higher dose gamma irradiated polyethylene film is not limited significantly by instrument sensitivity. The accuracy of dose determinations has been found to increase with dose, and is approximately ± 5 percent at 4.7×10^7 rads. It is believed that the upper gamma dose level for activation dosimetry will extend above 5×10^8 rads. This assumption is based upon the recent finding that the oxygen concentration in irradiated film has been found to increase with dose up to 5×10^8 rads, which was the largest dose studied. These latter analyses were performed using the classical Unterzaucher technique. Deterioration of polyethylene film caused by gamma radiation does occur but does not restrict the use of polymer films for gamma dosimetry, when using the activation technique.

CONCLUSIONS

This report describes a gamma dose measurement technique which uses a polymer detector. The technique demonstrates the feasibility of utilizing fast neutron activation analysis as a tool for determining the gamma dose received by a polyethylene film. This technique requires that the polymer detector be irradiated in air. The oxygen uptake of the polymer detectors, by chemical reaction, is induced by ionizing radiation and is related to gamma dose. The determination of dose is performed subsequently, by correlation of the fast neutron induced activity of oxygen in the polyethylene films, with previously established calibrated gamma doses. The results have shown that the oxygen-16 activation technique used in conjunction with the polymer detector offers several advantages for gamma dosimetry use in the megarad region.

In addition it is shown that the oxygen content of low density polyethylene film can be determined by fast neutron activation analysis. It should be noted that the technique should in no way be limited to use with gamma irradiated polyethylene, but may be used to determine the oxygen content of any polyethylene regardless of the origin of the contained oxygen. It is also evident that the polyethylene films did react with sufficient atmospheric oxygen to be analyzed quantitatively for oxygen by neutron activation. The infrared spectra of the irradiated films show that the radiation induced oxygen content is present primarily in the form of carbonyl and hydroxyl groups.

As the course of the activation experiments progressed, the results indicated the potential usefulness of the technique for high level dosimetry. It became evident also, that other areas should be studied and some additional aspects amplified. Thus, it would be desirable to: (1) establish the optimum geometry and mass of the polymer detectors for maximum gamma sensitivity and widest useful operating range; (2) establish the overall accuracy of the optimized system; (3) investigate the gamma energy and rate dependence of the system; (4) establish the sensitivity of the system to other types of ionizing radiation; (5) investigate the response of the polymer detectors in pulsed and steady state mixed radiation fields, and (6) study polypropylene as an air irradiated gamma detector film for use with activation analysis.

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It is highly desirable that the abstract of classified reports be unclassified. Each paragraph of the abstract shall end with an indication of the military security classification of the information in the paragraph, represented as (TS), (S), (C), or (U).

There is no limitation on the length of the abstract. However, the suggested length is from 150 to 225 words.

14. **KEY WORDS:** Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, rules, and weights is optional.